

CITRUS

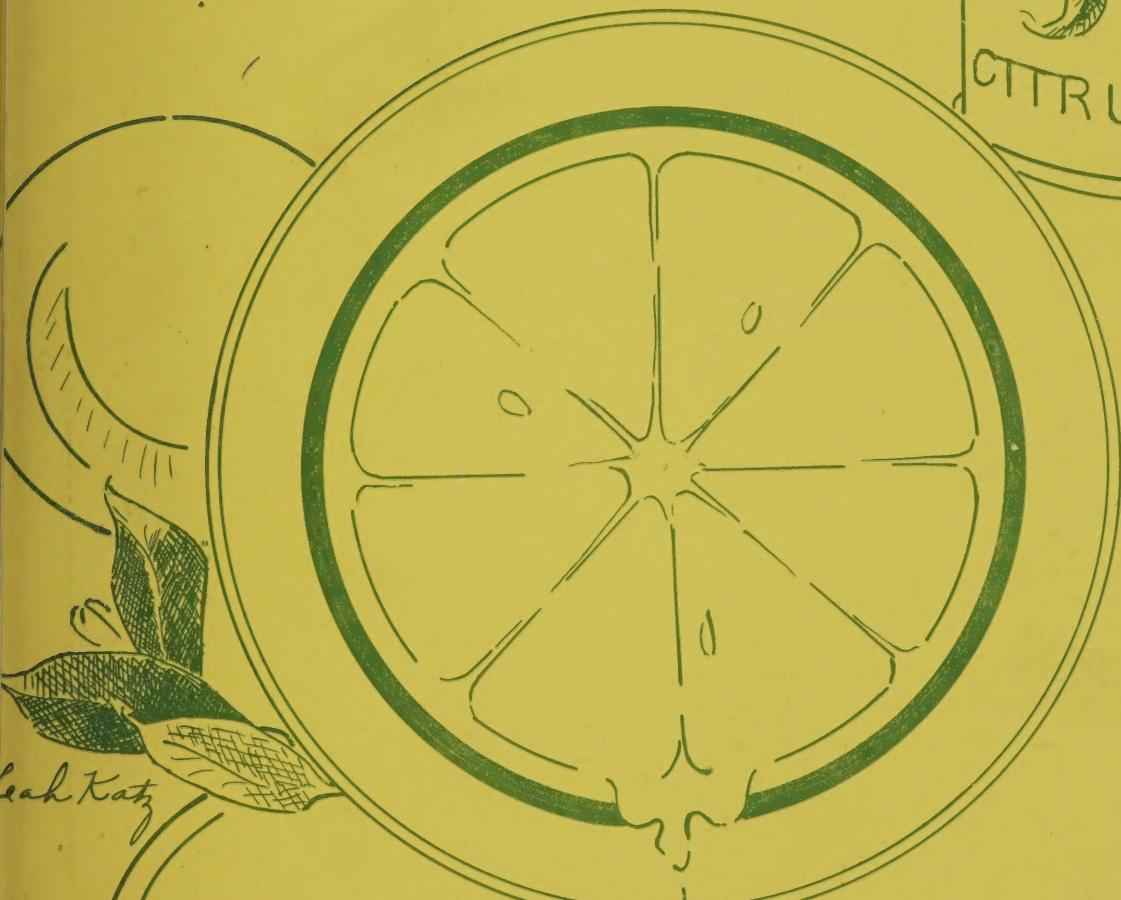
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Processing Conference

October 1, 1957



CITRUS JUICE



UNITED STATES DEPARTMENT OF AGRICULTURE

Agricultural Research Service

Southern Utilization Research and Development Division

U. S. Citrus Products Station

Winter Haven, Florida

PROGRAM AND ABSTRACTS OF PAPERS

SEVENTH CITRUS PROCESSING CONFERENCE

October 1, 1957

Florida Room, Citrus Building
Winter Haven, Florida

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ORGANIZATIONS PARTICIPATING IN
SEVENTH CITRUS PROCESSING CONFERENCE

SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION
Citrus Products Station, Winter Haven, Florida
Fruit and Vegetable Products Laboratory, Weslaco, Texas

WESTERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION
Fruit and Vegetable Chemistry Laboratory, Pasadena, Cal.

UNIVERSITY OF PUERTO RICO AGRICULTURAL EXPERIMENT STATION
Food Technology Laboratory, Rio Piedras

PROGRAM --- CITRUS PROCESSING CONFERENCE
(October 1, 1957)

MORNING SESSION - 9:30 A.M.

(M. K. Veldhuis, In Charge, Citrus Products Station, Winter Haven, Florida; Presiding)

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AFTERNOON SESSION - 1:30 P.M.

(V. H. McFarlane, Head, Fruit and Vegetable Section,
Southern Utilization Research and Development
Division, New Orleans, Louisiana; Presiding)

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FURTHER PROGRESS IN RESEARCH ON THE BITTERNESS OF ORANGE PEEL

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Winter Haven, Florida

Bitterness in orange peel is of interest because of the possible influence on the flavor of citrus juices, concentrates, marmalades, and other citrus products. So far, little is known of the nature of this flavor. The principal flavonoid in oranges, hesperidin, is so insoluble that it is almost tasteless and it is thought that other constituents must be involved. The fact that the addition of only 3% of peel juice to ordinary orange juice can be detected demonstrates that the flavor is potent. During the past year studies on these bitter constituents have continued.

Treatment of peel juice with activated charcoal removed the bitterness but elution of the bitter substance was difficult. Later, it was found possible to extract the bitter substance from peel juice with carbon tetrachloride. If the juice were first made alkaline to hold the phenolic flavonoids in solution as salts, extraction of the bitter material was still possible, showing that it was neutral or only faintly acidic in nature. Fractional crystallization resulted in the isolation of nobiletin and another methylated flavonoid which, like nobiletin, gave veratric acid upon hydrolysis. However, the principal bitter materials remained in the uncyclizable mother liquors. Subsequently, it was learned that the bitter material could be steam-distilled from the mother liquors or from the original extract. This eliminated the flavonoids and probably some other interfering materials. The bitter substance obtained in this way was a viscous oil, dextro-rotatory, and heavier than water. Three 2,4-dinitrophenylhydrazones were isolated from this oil; one from a crystalline sodium bisulfite addition product, one from a soluble sodium bisulfite addition product, and one from the oil itself after the others had been removed. However, these carbonyl compounds appeared to constitute only a small part of the oil and hence probably did not include the principal bitter substance. No alcohols have been found but there is some evidence for believing lactones are present. A large-scale extraction was conducted. A new degradation product of nobiletin is tetramethoxy salicylic acid. Besides the interesting chemistry of its anhydride, it might have therapeutic properties.

Considerable interest has been shown in finding an objective test for excessive extraction and finishes pressures. Some of the efforts in this direction have employed staining techniques for suspended particles. One of the most promising ones tried in this laboratory makes use of a phloroglucinol-hydrochloric acid reagent which stains lignin particles red or purple. While this has not been used and has not been developed to its final form, progress on it will be described.

TROPICAL FRUIT UTILIZATION RESEARCH AT THE PUERTO RICO
AGRICULTURAL EXPERIMENT STATION

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Rio Piedras

Many tropical fruits such as mangos, guavas, soursop, tamarind, papaya, oranges, grapefruit, pineapple, bananas and plantains grow well in Puerto Rico. Although almost all these fruits are seasonal, their production is spread throughout the year. The availability of these fruits the year around offers a challenging opportunity for the operation of processing plants on a 12 month's schedule. Industrialization of many of these fruits, however, has been limited by the lack of information on processing methods and keeping quality of the processed products.

Studies conducted at the Puerto Rico Agricultural Experiment Station have shown that excellent nectars can be prepared from guavas, mangos and soursop. It has been found that the removal of stone cells from guava nectar is of paramount importance in the preparation of a high quality product. A method has been devised for the removal of the stone cells which involves a selective centrifugation of the dispersed fruit pulp. It has been found that a nectar can be prepared from mangos ripening yellow by dispersing the whole fruit in water by the use of a special high speed stirrer. By a similar method a nectar can be prepared from soursop. All these nectars keep very well at 85°F. and are fairly good sources of Vitamin C.

For the past five years, West Indian Cherries or Acerolas have attracted the attention of the food processor as the best source of natural Vitamin C. Extensive studies have been carried out to develop a method for canning the Acerola juice. The results obtained show that when the juice is pasteurized and canned, a rapid loss in color takes place which is accompanied by the production of carbon dioxide and swelling of the cans. This reaction proceeds at a very fast rate when the product is stored at room temperature (85°F.). The canned juice keeps fairly well at 45°F. without appreciable production of carbon dioxide. The frozen product keeps for a year without deterioration. Production of gas in the canned product is paralleled by a partial destruction of ascorbic acid and pigment loss. Carbon dioxide seems to be produced by the reaction of the ascorbic acid with malvidin, the main anthocyanin pigment present in the fruit. Complete removal of the pigment apparently improves the keeping quality of the juice.

Both single strength acerola juice and ten-fold concentrates have been found suitable for enrichening pear and peach nectar and apricot and grape juice. The addition of one part of the single strength juice to 27 parts of any of these products resulted in the increase of ascorbic acid from 1 mg. per 100 ml. to 60 mgs. No flavor change or development of off-flavors could be observed in the finished product.

It is well known that off-flavors develop rapidly in single strength hot packed orange and grapefruit juices. It has been found that the addition of pineapple juice of the Red Spanish variety to either orange or grapefruit juice or to a blend of both, prevents the development of off-flavors. Blends of two parts orange juice, one part pineapple juice and one part grapefruit were found to keep for 10 months at room temperature without appreciable development of off-flavors. A blend of two parts orange juice and one part pineapple juice was found to be equally stable under similar storage conditions.

The development of procedures for the production of fruit jellies and pastes has also been one of the objectives of our tropical fruit utilization research program. Emphasis has been given to the study of methods for the preparation and packing of guava jelly and paste. The suitability of different varieties of guavas both for jelly and paste manufacture has been studied. A method has been worked out for the determination of jelly strength using the Brookfield viscometer and path penetrometer. A marketing test will be conducted this year to test the suitability of different containers for packing guava paste such as aluminum ribfoil cups, tin cans and cardboard glassine-lined boxes.

During this year, at our new Food Technology Laboratory, studies on the freezing of tropical fruits are being initiated. Likewise, methods and procedures for the utilization of bananas and plantains are being studied. The banana and plantain utilization research program involves the preparation of hot-packed acidified banana puree and frozen puree prepared from those varieties of bananas of commercial importance to Puerto Rico. The production of plantain chips, canned and frozen plantains and dehydrated bananas and plantains are also being studied.

A REVIEW OF RESEARCH ON THE PRODUCTION AND STORAGE STABILITY OF FROZEN HIGH-DENSITY ORANGE CONCENTRATE

E. A. BEAVENS
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Pasadena, California

Basic compositional and pilot plant studies indicated that the flavoring components of orange juice lost during evaporation were mainly volatile oils and up to 95% of these oils found in the juice originated in the flavedo of the peel. On the basis of this information a procedure was developed for flavor fortifying concentrated orange juice which consisted of adding small amounts of high quality cold-pressed orange peel oil instead of the "cut-back" juice. Flavor fortified superconcentrates of 5-, 6-, or 7-fold concentration are thus obtainable as there is no dilution from added juice.

Taste panel tests of frozen orange superconcentrates flavor-fortified with added oil indicated their flavor and aroma were comparable to that of commercial 4-fold concentrate prepared by the "cut-back" process. Storage studies indicated that flavor stability was superior to either heated or unheated 4-fold concentrates. Also, it was found that cloud stability was greater than that of unheated 4-fold concentrates. In fact, it was comparable to that obtained by heating 4-fold concentrates at 150° - 160° F. for 1-20 seconds.

Frozen superconcentrates have certain desirable physical properties. They do not become solid on freezing, a feature of interest to handlers of institutional-size containers because of the shorter time required for reconstitution. The volume of 4-fold concentrate increases about 5% on freezing, whereas superconcentrates show no appreciable volume change so containers may be more completely filled. Superconcentrates begin to freeze at approximately 5° F. and contain about 2.5% ice at 0° F., while 4-fold concentrates begin to freeze at 17.6° F. and contain 32% ice at 0° F. These factors might aid in saving container costs, refrigerated storage space, and shipping costs as there is less water to freeze and keep frozen and less to be shipped.

^{1/} Western Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

RECENT ADVANCES IN THE STUDIES OF THE CHEMICAL COMPOSITION OF
LEMON OIL AND THE FLAVONOID CONSTITUENTS OF THE LEMON

W. L. STANLEY AND R. M. HOROWITZ
Fruit and Vegetable Chemistry Laboratory^{1/}
Pasadena, California

Investigations are continuing on the coumarin and psoralen compounds in lemon oil. Ten compounds have been isolated to date of which three are coumarin ethers and seven are furocoumarin ethers (psoralen ethers). The identity of five of the compounds has been established and the identity of the other five tentatively established. Examination of other citrus oils has revealed unique differences in the distribution of these compounds. From this information and a knowledge of the spectral absorption characteristics and migration rates (R_f values) of the compounds methods of analysis have been developed for determining their presence in lemon oil both qualitatively and quantitatively. Concurrently it has been possible to demonstrate the application of the methods to the determination of mixtures of other citrus oils in lemon oil.

A new specific reagent system has been discovered for citral (the characteristic odorant of lemon oil) and dihydrocitrinal. This reagent system has been developed into a quantitative colorimetric procedure accurate to concentrations below 0.1% citral.

A new flavonol which has been named limocitrin has been isolated from hydrolyzed extracts of lemon peel. Degradative and spectroscopic studies show the structure to be 3', 8-dimethoxy-3,5,7,4'-tetrahydroxy-flavone. This is the first example of a derivative of gossypetin occurring in citrus. In addition, luteolin (5,7,3',4'-tetrahydroxy-flavone) has been isolated and identified from hydrolyzed extracts of lemon peel.

^{1/} Western Utilization Research and Development Division,
Agricultural Research Service, U. S. Department of Agriculture.

PROGRESS ON THE NEW "FRUIT AND VEGETABLE PRODUCTS LABORATORY"
BUILDING AT WINTER HAVEN

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Citrus Products Station
Winter Haven, Florida

At the time of the Citrus Processing Conference last year, the appropriation for the new building had been passed by Congress and the General Services Administration was engaged in selecting a private architectural firm to design the new building and prepare the specifications. Shortly after the meeting, the firm of James Gamble Rogers II, Winter Park, Florida, was selected.

During the winter, the plans began to take shape. There were several stages and each had to be approved and an estimate of the cost prepared before proceeding to the next. Final plans were submitted by the architects to the General Services Administration in Washington early in April. Sealed bids were opened at the General Services Administration Office, Atlanta, Georgia, on June 11. Paul Smith Construction Company of Tampa was the low bidder and received the contract. Actual work at the site began on July 24, 1957. The contract states that the building is to be completed within 365 calendar days, so we can expect to occupy the new facilities next summer and should be settled by the time of the next conference.

The building will be split level with the laboratory floor and basement on the north side and the pilot plant on the south. The walls will be of reinforced concrete or concrete blocks and will be faced with light red brick. It will be located on Avenue S N.W. between 6th and 7th Streets on a site provided by the City of Winter Haven. The building will face North on Inman Park which is directly across U. S. Highway 17 from the Howard Johnson Restaurant. The site is considered to be an excellent one.

Most of the facilities illustrated in earlier floor plans were obtained in the building. Shortly after the appropriation was passed, there was a substantial increase in the price of steel and other items and it was necessary to reduce the total floor space somewhat, but this was done with the loss of a minimum of essential facilities.

The main laboratory floor will be air conditioned as well as a few rooms in the basement. The system is so arranged that there is no general recirculation of air, and odors will not be carried from one room to another. Each room is supplied with sufficient cooled air for ventilation and where added cooling is required, a separate cooling unit is installed.

There are to be nine main laboratories measuring 16 x 29 feet and in addition a chromatographic laboratory, oven room, sensory evaluation laboratory, instrument room, preparation room, first aid room, drafting room, dark room and several offices and storerooms. In the basement there are to be constant temperature rooms that can be operated from -10° to 80° F. The pilot plant will be 132 feet long by 48 feet wide and will have 16 feet clearance to the bottom of the roof trusses.

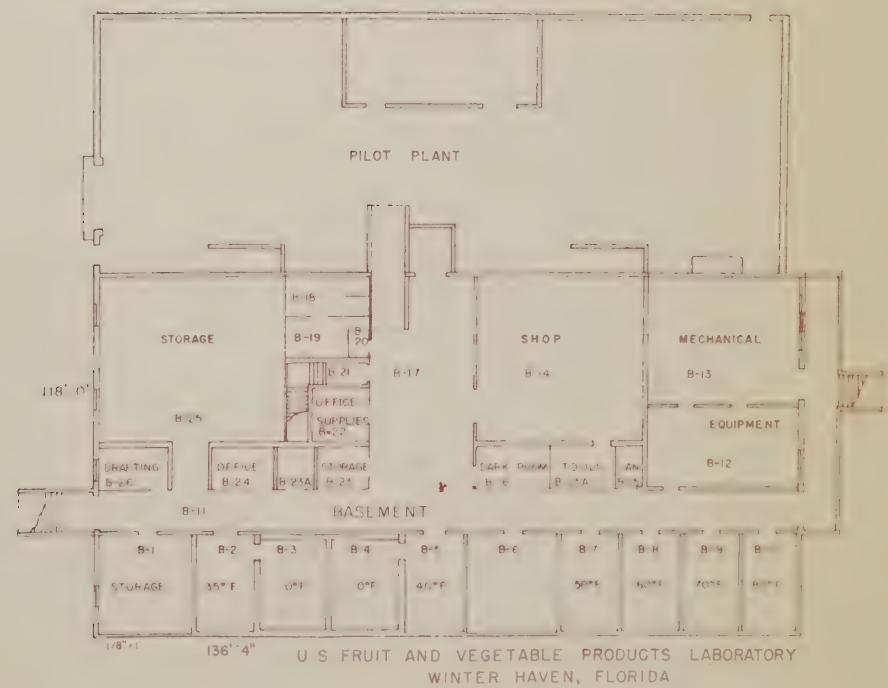
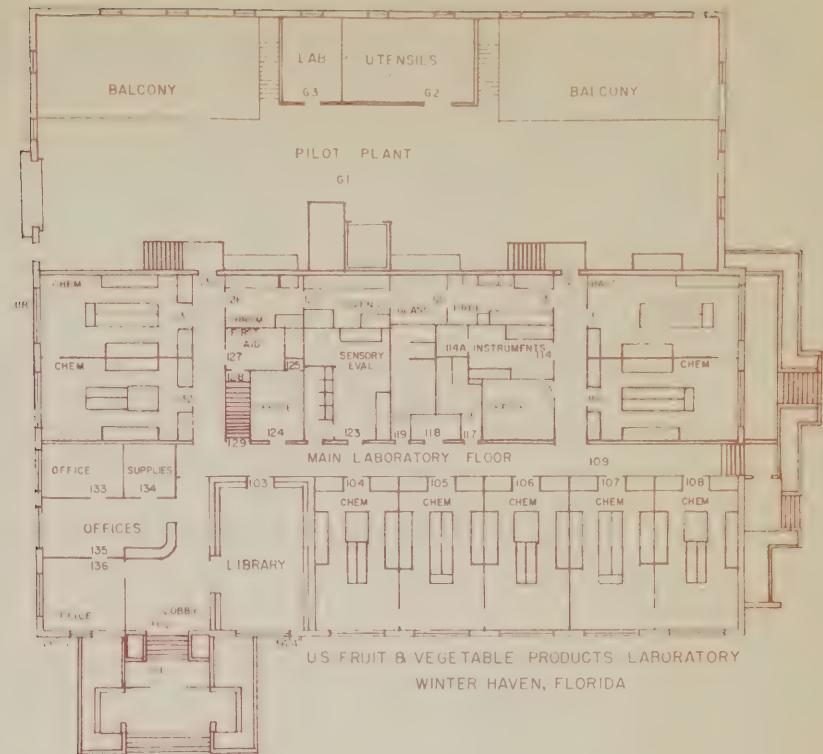
Along one side of the pilot plant there is to be a balcony which will increase the floor area. Also included in the plans is a solvent storage house 15 feet by 15 feet for the storage of inflammable materials. A four-car garage is provided.

The plans call for the installation of all permanent laboratory equipment such as workbenches, hoods, sinks, dishwashers, autoclaves, and the like. New desks and other office furniture will be provided. In the pilot plant, a system is to be installed for handling the vapors from the vacuum evaporator. Some pilot plant equipment will be transferred from the present laboratory and other equipment will be added as needed. Usable laboratory equipment will also be transferred.

As is indicated by the high proportion of laboratory space, much emphasis in the new facilities will be given to fundamental problems. The pilot plant will be needed to provide materials of various kinds for use in the laboratory and will also provide a place to try out new ideas developed in the laboratory.

The principal emphasis will be on new and improved citrus products, but as the new name indicates, consideration will be given to other fruits and vegetables that show promise.

A copy of the floor plan is included in the proceedings of the conference.



REPORT OF PROGRESS ON CITRUS RESEARCH AT THE
U. S. FRUIT AND VEGETABLE PRODUCTS LABORATORY, WESLACO, TEXAS

F. P. GRIFFITHS
Fruit and Vegetable Products Laboratory
Weslaco, Texas

Research on citrus at the Texas laboratory is being carried forward under several line projects. These are concerned with the:

Biochemical investigations of the progressive changes in the carotenoid pigment content of colored grapefruit during the growing season as related to color characteristics and quality of processed juice products.

Development of improved processing procedures for the utilization of poorly colored and late season red and pink grapefruit.

Investigations to reduce the bitterness and enhance the quality of grapefruit juice products. (Joint project with Winter Haven laboratory.)

Investigations of the processing characteristics of red grapefruit as affected by variety of rootstocks to facilitate the selection of rootstock for production of fruit giving processed products of optimum quality. (In cooperation with Dr. Cooper, In Charge, Citrus Rootstock Investigation Laboratory.)

Investigations of minor fruits.

Carotenoid Pigments in Grapefruit. Biochemical investigations of the development of color in white, pink and red grapefruit are still incomplete. Work on separation of the minor pigments from carotene and lycopene has led to the development of an improved method of partition chromatography. A silica gel column treated with methyl alcohol is used to separate hydrocarbon carotenoids such as carotene and lycopene from monohydroxy and polyhydroxy carotenoids. This technique permits the separation of the minor pigments in sufficient quantity for further study. It is a much more rapid procedure than counter-current phase separation. Details of the procedure will be published soon in the Journal of Analytical Chemistry.

Pulp-Fortification of Canned Juice. Work on the canning of red grapefruit juice has demonstrated that color, flavor and nutritional value can be enhanced by additional pulp in the juice. Difficulties were encountered in obtaining pulp free of seeds and seed fragments. Washing and floating the pulp did not completely remove seeds. A method of recovering seed-free pulp has been devised which appears to be practical.

A finisher is equipped with brushes instead of paddles and waste pulp from the regular finisher re-run through this. The brushes work a large percentage of cellular fragments through the screen without including seeds or seed fragments. Pulp from a regular finisher yielded about one-half as much seed-free material of good color and about 80% suspended solids content. Recovered pulp was preserved by canning (pasteurization) and by freezing, and stored for addition to poorly colored late season juice. Good color retention was obtained in pulp which was flash pasteurized, canned, and held at room temperature, and in pulp which was placed in sealed containers and kept frozen at 0° C. Addition of from 5 to 10% by volume of highly colored pulp to juice having a "faded" appearance, materially improved the product.

The methods for pulp preservation have been investigated on a laboratory scale and in the 1957-58 season an endeavor will be made to check them on a pilot plant or semi-commercial scale.

Centrifuging poorly colored juice and replacement of pulp with pulp from white fruit is another method which has been tried for improving juice obtained from late season "faded" colored fruit.

Reduction of Bitterness in Grapefruit Juice Products. Because highly colored pulp from early season fruit contains more naringin than is desirable and bitterness may limit its addition to juice, investigations were continued on methods of reducing the bitterness of pulp and juice. Washing the recovered pulp in warm water for about 3 minutes reduces naringin content without altering color. Acid, sugar and fruit flavors, as well as naringin, are removed by washing. A better approach appeared to be a study of enzyme hydrolysis of naringin in the juice and pulp. S. V. Ting (Citrus Experiment Station, Lake Alfred) in 1955, reported the use of an enzyme contained as an impurity in Pectinol to debitter juice and pulp. Small amounts of purified enzymes, naringinase A and B, were supplied to the U. S. Fruit and Vegetable Products Laboratory by Rohm and Haas Company, Philadelphia, for experimental purposes.

Experiments have shown that pulp can be sufficiently debittered by 0.1% of this enzyme in 1/2 to 1 hour at 40° C. without affecting its color. Debittered pulp has been pasteurized to inactivate the enzyme and added to juice. Color was enhanced without adversely affecting cloud or flavor of the fortified juice. Juice containing more than the desired amount of naringin has been debittered to acceptable levels without affecting cloud or flavor. Enzyme was also successfully used to remove bitterness of grapefruit peel used for marmalade.

Paper chromatographic studies on the enzymatic hydrolysis of pure naringin show rapid and simultaneous appearance of the sugars, rhamnose and glucose, in the hydrolysate. Good correlation between observed bitterness and the Davis test for flavonoid content of juice before and after enzymatic hydrolysis was not obtained. Enzyme hydrolysis may change the Davis reaction of other flavonoids in grapefruit juice and may not be specific for naringin. Experiments indicate that during

the first stages of enzyme hydrolysis bitterness decreases more than the Davis test indicates.

All data on the enzyme experiments need confirmation and application on a larger scale. The results to date appear promising. Should a practical method evolve for the enzyme debittering of citrus juices and pulp, a cheap source of enzyme will be needed.

Effect of Rootstock on Processing Quality of Red Grapefruit. In cooperation with Dr. W. C. Cooper and staff of the Citrus Rootstock Investigations Laboratory of the Horticultural Crops Research Branch, ARS, studies are continuing on the effects of different rootstocks on the processing qualities of Ruty red grapefruit. Fruits from trees on 10 or 12 of what appear to be the best rootstocks are harvested four times during the season and studied to determine existing differences. Size, weight, rind thickness and color of the fruit is noted, and acid, Brix, naringin content and taste is determined on the pulp and juice from fruit of each sampling. Differences in characteristics of fruit from different rootstocks have been noted in prior season's data and it is apparent that several years' more work will be necessary to establish the significance and constancy of observed differences.

Meyer Lemon Juice Concentrate for Lemonade. Work has been completed on the use of Meyer lemon juice for preparing concentrate for lemonade. It has been shown that a more acceptable concentrate for lemonade can be prepared from Meyer lemons if a small amount of oil from a variety such as Eureka lemons (USP Oil of Lemon) is added to the juice-sugar mixture before freezing. The experimental data are in preparation for publication.

USE OF THE REFRACTOMETER FOR MEASURING SOLIDS IN CONCENTRATED CITRUS JUICES

M. K. VELDHUIS, D. A. MORGAN AND W. C. SCOTT
Citrus Products Station
Winter Haven, Florida

I. BACKGROUND INFORMATION (M. K. VELDHUIS)

Last year we presented information on the effect of various known constituents of concentrated citrus juices on their refractive index. Their quantitative effects were determined by measuring the refractive index of the pure constituents in water solution and in sugar syrups. Charts were shown which indicated the size of corrections needed for the presence of these constituents in proportions in which the literature indicated they might be found in citrus juices.

Since our last report, we have analyzed a series of 50 samples of commercial and laboratory samples of frozen concentrated orange, grapefruit, and tangerine juices and 2 samples each of concentrate for lemonade and for limeade, for those constituents found to seriously affect the accuracy of the refractometer when used to determine soluble solids. Results of these analyses will be presented by Mr. Morgan and Mr. Scott. Mr. Morgan will then discuss some additional work he did on the effect of pulp on the refractive index of sucrose syrups, and Mr. Scott will summarize the cumulative effect of correcting refractometer readings for the various constituents.

II. SOLUBLE NITROGEN AND MINERAL CONTENT OF CONCENTRATES (D. A. MORGAN)

Amino acids have been reported to exist in orange juice in proportions as high as 7.5% of the total solids. Refractometer readings of amino acid solutions are higher on the sucrose scale than their actual soluble solids content, and negative corrections required are relatively large as compared to the other constituents of citrus juices. A study of the amino content of concentrates was therefore essential.

Since the study of the amino acid composition of Florida citrus fruits would be a major study of itself, it was decided that the determination of actual amino acids of the series of samples under study would not be undertaken. It has been reported that 80-90% of the soluble nitrogen in citrus juices exists as free amino acids and related compounds. Therefore, for the purposes of this study, it was considered adequate to determine the soluble nitrogen content of the samples, convert that to amino acids by an appropriate factor, and assume that the nitrogenous materials thus estimated would approximate the effect on refractometer readings of an equivalent solution of mixed amino acids. The ranges of amino acid equivalents, based on the soluble solids of each concentrate, are shown in Table I.



TABLE I

Range of amino acid equivalents

Samples		Percent soluble solids (dry basis)			
Type	No.	High	Low	Ave.	Std. Dev.
Fla. orange	32	5.0	3.2	4.0	0.46
Pinellas Co. (Oct.)	1			2.0	
Fla. grapefruit	11	4.8	3.2	4.2	0.63
Fla. tangerine	4	3.9	3.5	3.7	
Calif. blood org.	1			3.9	
Calif. concentrate	1			6.7	
Fla. lemonade	2			Tr.	
Fla. limeade	2			Tr.	

The effect of these quantities of amino acids on refractometer readings is shown in Table II.

TABLE II

Range of refractometer corrections for amino acids

Dry basis		40° Brix		60° Brix	
% Protein	% Protein	Correction	% Protein	Correction	
High	5.0	2.0	-0.555	3.0	-0.830
Low	3.2	1.3	-0.360	2.0	-0.555
Ave.	4.0	1.6	-0.445	2.4	-0.665
Maximum variation from average		.11			.165

From these figures it is apparent that, in order to obtain results more accurate than 0.2° in the use of the refractometer, it would be necessary to analyze each sample for amino acid content.

The effects of mineral constituents on refractive index were shown last year to be significant. These are most prevalent in the form of metallic citrates. In order to arrive at an estimate of the mineral content of the series of samples, it was necessary to determine the ash and potassium content of the clarified juices. The range of ash and mineral constituents found are shown in Table III.

TABLE III

Constituent	Range of mineral constituents of concentrates			
	High	Low	Ave.	Std. Dev.
Orange juice				
Ash (% of solids)	4.30	3.25	3.87	0.27
K ₂ CO ₃ (% of ash)	86.2	76.2	80.7	2.5
K-Citrate (% of solids)	7.65	5.80	6.94	0.40
Grapefruit juice (unsweetened)				
Ash (% of solids)	4.18	2.81	3.51	0.45
K ₂ CO ₃ (% of ash)	84.6	72.5	80.6	3.7
K-Citrate (% of solids)	7.65	4.90	6.26	0.77

The mineral constituents of unsweetened tangerine concentrates varied but little from those of orange and grapefruit. Sweetened grapefruit and tangerine concentrates had slightly lower mineral content.

Analyses of the ash of concentrated citrus juices by the Wisconsin Alumni Research Foundation indicate a fairly constant relationship between potassium, sodium, calcium, and magnesium. Using their average figures, it was possible to estimate, from the potassium content as found, the probable calcium, magnesium, potassium, and sodium citrate contents of each of the samples. The range of total citrate salts, and refractometer corrections therefor, are shown in Table IV.

TABLE IV

Range of refractometer corrections for citrate salts						
	Dry basis	40° Brix		60° Brix		
	% Ash	% Salts	% Salts	Corr.	% Salts	Corr.
Concentrated orange juice						
High	4.30	9.35	3.74	-0.301	5.60	-0.403
Low	3.25	7.07	2.83	-0.250	4.24	-0.328
Ave.	3.87	8.41	3.36	-0.281	5.04	-0.371
Concentrated grapefruit juice (unsweetened)						
High	4.18	9.08	3.63	-0.302	5.45	-0.392
Low	2.81	6.11	2.44	-0.225	3.67	-0.303
Ave.	3.51	7.64	3.05	-0.262	4.58	-0.352
Maximum variation from average				0.040		0.049

Since the maximum variation from the average of both orange and grapefruit concentrates is within the limits of accuracy of reading the refractometer (.05° Brix), it appears likely that the use of a standard correction for mineral content of concentrate of a given concentration would be satisfactory. The above results indicate the subtraction of 0.04° Brix from the refractometer reading of 42° Brix concentrate might be reasonable.

III. PULP, SOLUBLE SOLIDS AND SUGAR CONTENT OF CONCENTRATES (W. C. SCOTT)

In examining this series of concentrates for pulp, the Alcohol-Acetone (A-A) insoluble solids method described at the 1956 Conference was used. It was found that the ratio of water insolubles to A-A insolubles was fairly constant at about 0.56:1. Also described was the vacuum oven method for determining total solids. Soluble solids are recorded as the difference between total solids and water insolubles (56% of the A-A insolubles). The determination of the effects of pulp on refractometer readings of sugar solutions is quite involved, and will be covered in a separate report.

The reducing sugars in citrus juices, fructose and glucose, have lower indices of refraction than sucrose, therefore additive corrections must be made for their presence. Fructose, glucose, and sucrose were determined by reduction of alkaline ferricyanide, and colorimetric estimation of resultant ferrocyanide as described by Ting. The ratios of sugars found in concentrated orange juices agree fairly well with results reported by Curl and Veldhuis in 1948, as shown in Table I.

TABLE I

Samples		Ratios of sugars in concentrated orange juices			
Type	No.	Glucose Red. sugar x 100	Red. sugar Total sugar x 100	Total sugar Refrac(uncorr.) x 100	
Early season	14	Max. 53.9	51.0	88.5	
		Min. 42.5	41.5	76.6	
		Ave. 48.7	46.6	80.6	
Mid season	12	Max. 51.9	51.5	86.2	
		Min. 43.4	42.9	73.7	
		Ave. 49.4	47.9	79.9	
Late season	13	Max. 51.9	50.8	82.6	
		Min. 46.5	45.8	73.7	
		Ave. 49.1	48.7	78.9	
Valencia, 1948		48.0	49.5	79.0	
Calif. conc.		49.2	53.5	73.3	

The ratios of sugars found in grapefruit concentrates are somewhat different, as shown in Table II.

TABLE II

Ratios of sugars in concentrated grapefruit juices

Samples		Glucose	Red. sugar	Total sugar
Type	No.	Red. sugar x 100	Total sugar x 100	Refrac(uncorr.) x 100
Unsweetened	6	Max. 69.3	61.2	86.4
		Min. 48.6	56.6	78.1
		Ave. 56.9	58.9	81.0
Sweetened	4	Max. 52.8	54.4	83.9
		Min. 51.4	39.9	82.2
		Ave. 52.3	46.4	83.2

The ratios of sugars in tangerine, lemon and lime concentrates are shown in Table III.

TABLE III

Ratios of sugars in tangerine, lemon and lime concentrates

Samples		Glucose	Red. sugar	Total sugar
Type	No.	Red. sugar x 100	Total sugar x 100	Refrac(uncorr.) x 100
Unsweetened tangerine	2	53.9	42.1	83.3
		48.7	41.3	80.0
Sweetened tangerine	2	49.8	40.8	81.9
		44.6	37.1	79.1
Conc. for lemonade	2	83.9	4.1	79.8
		57.6	3.7	95.5
Conc. for limeade	2	52.6	2.7	98.6
		48.7	37.2	98.0

An idea of the magnitude of corrections made necessary by the presence of reducing sugars in concentrated orange juice may be obtained from data shown in Tables IV and V.

TABLE IV

Corrections for fructose in concentrated orange juice

	Calculated to 42° Brix		Calculated to 58.5° Brix	
	Percent	Correction	Percent	Correction
Maximum	8.60	+0.096	11.98	+0.146
Minimum	7.40	+0.079	10.30	+0.121
Average	8.17	+0.090	11.36	+0.136
Max. Dev. from Ave.	0.77	+0.011	1.06	+0.016
Std. Dev.	0.294	(0.005)	0.433	(0.006)

TABLE V

Corrections for glucose in concentrated orange juices

	Calculated to 42° Brix		Calculated to 58.5° Brix	
	Percent	Correction	Percent	Correction
Maximum	8.87	+0.044	12.36	+0.072
Minimum	6.13	+0.022	8.54	+0.042
Average	7.80	+0.036	10.50	+0.057
Max. Dev. from Ave.	1.67	+0.013	1.96	+0.016
Std. Dev.	0.670	(0.005)	0.916	(0.008)

From the data presented in Tables IV and V it can be deduced that, for the Florida orange concentrates studied and reduced to 42° Brix by calculation from their uncorrected refractometer values, a correction for reducing sugars of +0.126° was within 0.005° of the correct figure two-thirds of the time. For the same series of concentrates calculated to 58.5° Brix, a correction of +0.193 was within 0.007° of the correct figure two-thirds of the time.

Corrections for reducing sugars in the other citrus concentrates vary only slightly from those required for orange concentrate.

IV. EFFECTS OF PULP ON REFRACTOMETER READINGS OF SUCROSE SYRUP (D. A. MORGAN)

This series of experiments was designed to determine the effect of pulp on refractometer values. In most of the work, pulp was obtained by centrifuging fresh single strength orange juice. This pulp, or centrifuge mud, was assumed to be wet with juice whose gross composition in regard to soluble solids could be readily determined, and its moisture content was determined by the Karl Fischer method. It was then added in measured amounts to sucrose solutions of various concentrations, and changes in refractometer readings recorded. Differences were found in percentage of change at the different sugar concentrations. When the pulp was washed in order to remove water solubles, then added to sugar syrups, still different effects were obtained. Also,

when pulp was washed either with water or alcohol-acetone and dried, then added to the syrups, effects differed from both raw pulp and washed pulp. Freezing of the pulp, either while still on the tree or in concentrate, changed its effect on refractometer readings. The outstanding similarity between all the tests made so far is that the addition of pulp increases the refractometer reading more than could be accounted for by mere mechanical dilution.

In spite of the above anomalies, sufficient data has been accumulated to justify the use of a single curve in estimating correction factors for concentrates of varying pulp content and at different concentrations when prepared from normal fruit (not frozen or showing excessive dryness).

V. INDIVIDUAL AND CUMULATIVE EFFECTS OF CONSTITUENTS ON REFRACTOMETER READINGS (W. C. SCOTT)

Citric acid, fructose, and glucose have refractive indices lower than that for sucrose of equivalent concentration, therefore a positive correction is required for the refractometer sucrose scale to accurately reflect the true soluble solids content of their solutions. Amino acids and mineral salts have refractive indices higher than sucrose, and a negative correction is required. Although pulp is insoluble and should not affect refractive index directly, it causes the refractometer to read erroneously high, and thus necessitates a negative correction. Upon totaling the three positive corrections and the three negative corrections experimentally determined, it was found that the net correction for the constituents studied was always negative and frequently as great as -0.5° Brix. After application of this negative correction, results with these samples indicate that there must still be unrecognized factors affecting refractometer readings, as the corrected readings still average nearly 1° Brix higher than soluble solids found by vacuum oven drying.

Uncorrected refractometer readings were found to more closely approach actual total solids content of concentrates than the corrected readings approach true soluble solids. Uncorrected refractometer readings usually were slightly higher than total solids and corrected readings were slightly lower. Corrected readings were usually closer to total solids than to soluble solids.

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List of Citrus Publications---January 1, 1953
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SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

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